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Development and Applications of Optical Interferometric Micrometrology in the Angstrom and Subangstrom Range

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DEVELOPMENT AND APPLICATIONS OF OPTICAL INTERFEROMETRIC MICROMETROLOGY IN THE ANGSTROM AND SUBANGSTROM RANGE

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SUMMARY

The recent development of the scanning electron tunneling microscope and the atomic force microscope requires absolute standards for measurements in the angstrom and subangstrom range. Optical interferometry with lasers and multiple mode laser resonances can provide absolute measurements as the laser wavelengths are very accurately known. A key feature of such measurements is the use of piezoelectric crystals as translators of the highest accuracy for very small distances. However, the dimensional changes of these crystals resulting from electrical potential changes depend on many variables, among them the method of mounting, so that accurate calibrations are necessary.

Starting from advances in optical metrology made by physicists trying to find gravity waves, advances which led to measurements down to 10^{-5} Å, the author designed and built a much simpler system for the angstrom range. The major limiting factors were mechanical vibrations, air currents, thermal changes and laser instabilities.

INTRODUCTION

The atomic theory goes back to the Greeks, but since atoms are too small to be seen there always remained a lingering doubt about their existence. Are they "real" or just convenient theoretical concepts? A solid surface is seen as impregnable, but if it consists of atoms spaced apart in regular or not so regular lattice configurations, then it is mostly open and only appears to be continuous, perhaps on account of rapid motion of the elementary particles. X-ray diffraction provides evidence of lattice structures of solids and of spacings between the "atoms" of the order of angstroms. The diffraction patterns show only the Fourier transforms of the real lattice or so-called reciprocal lattices, and therefore are still only constructs. Moreover, x-rays generally cannot provide surface maps. Using multiple beam optical interferometry Tolansky was able to show that some flat surfaces of crystals such

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as quartz or mica can contain steps and that organic crystalline materials, such as polyethylene of high density, can have a surface structure of planes and steps between them, that are of molecular dimensions. He produced surface maps by noncontacting techniques; however, resolution was atomic or molecular, i.e., a few angstroms, only in the direction normal to the surface. His lateral resolution was orders of magnitude poorer and he could not "see" the atoms or molecules on the surface.

Low energy electron diffraction techniques again do not permit the "seeing" of atoms and, moreover, are suspect because of the interaction of "foreign" bombarding electrons with the electrons surrounding the atoms of the surface: The bombardment, though of low energy, must still interact with the surface. Electron diffraction techniques, moreover, require long-range order in the samples. These techniques have also relatively poor lateral resolution.

The announcement of the invention and successful operation of a scanning electron tunneling microscope primarily useful for electrically conducting surfaces, and, a little later, of an atomic force microscope, potentially applicable to almost any surface, were therefore immediately greeted as major breakthroughs. These instruments can provide the lateral resolution necessary to provide atomic surface maps. Optical techniques are still needed to calibrate these new tools, but even the most sensitive optical techniques alone cannot provide the lateral resolution. The work described in this paper deals with the optical techniques considered appropriate for these calibrations, but they can be of use elsewhere. As a matter of fact, some of the optical techniques were developed in connection with the search, so far in vain, for gravity waves and can have extremely high sensitivities. One of these ultrasensitive techniques, fluorescence excited by evanescent waves, was never used for metrology but is likely to find a place, and is therefore included in this paper. The technique adapted for our experimental calibration setup, a variation of laser interferometry, turned out to be adequate and well-suited for the purpose.

For completeness a brief review of the new surface analysis tools precedes the discussion of optical techniques.

SCANNING TUNNELING MICROSCOPE AND ATOMIC FORCE MICROSCOPE

Scanning Tunneling Microscope

The scanning electron tunneling microscope (STM) of Binnig and Rohrer (refs. 1 and 2), developed only a few years ago, has excellent lateral resolution and does not affect the surface which it scans with atomic resolution. It provides surface maps of the electron densities as a function of potential. High concentrations of electrons correspond to the clouds surrounding atoms. The STM is basically very simple: A very sharp metallic tip is held at a constant small distance (subnanometer) above the test surface moving in the x or y direction. Electrons tunnel across the gap into the surface if the tip is biased negatively with respect to the surface, or vice versa if the bias is reversed. The bias potential is very low, from millivolts to a few volts. The tunneling electrons indicate the local electron densities in terms of changes in the tunneling current, which depends exponentially on the tip-to-surface

separation, and is typically an order of magnitude for every angstrom of separation. By scanning the tip across the surface an image in the lateral direction is achieved, whose resolution depends on the size and shape of the tip (2 to 3 Å). The vertical resolution depends on the mechanical and electronic stability of the instrument and is now typically 0.1 Å. Horizontal scanning is carried out with X- and Y-piezoelectric translators, while the tip is held at a constant distance from the surface by a third piezoelectric device.

The tunneling is a "mysterious" way by which electrons flow through the gap between the tip and the surface even through a vacuum. It can be explained only by quantum mechanics, where electrons are represented as electromagnetic waves and, just as light, can traverse a vacuum. Physically tunneling is readily differentiated from ordinary electron flow by the exponential dependence on gap width. An analogy to electron tunneling is light tunneling between two parallel planes when incident at angles greater than critical. This is taken up in a later section.

The ability of the STM to obtain the high horizontal and vertical resolution mentioned has been attributed to four main features (ref. 4):

(1) Piezoelectric transducers can achieve resolution better than 0.01 nm over a range of several micrometers.

(2) The mechanical drift rates attainable (less than 1 nm/min) are insignificant or can be corrected for over the typical image acquisition time of 1 sec to 5 min.

(3) Vibration isolation prevents building vibration and acoustic noise from affecting the sample-tip assembly.

(4) The rapid variation of the tunneling current with distance localizes the current at the very end of the tip.

Atomic Force Microscope

The most recent method today of seeing atoms on surfaces is the atomic force microscope (AFM) of Binnig, Quate, and Gerber (ref. 3). This instrument senses minute (10^{-10} to 10^{-8} N) forces between a sharp tip and a sample surface. It provides nondestructive surface profilometry at a resolution of better than 10 nm, and perhaps down to the atomic level. No stylus profilometer can match this performance. The AFM makes use of the first three features of the STM listed above but detects the force between the tip and the surface by the deflection of the cantilever on which the tip is mounted. This deflection can be measured by means of an STM attached to the same cantilever. It can also be measured by optical interference (ref. 4). As pointed out in reference 4, the AFM goes beyond the capabilities of simple profilometry, because it can quantitatively measure physical, chemical, magnetic, frictional, and electrostatic interfacial forces with very high spatial resolution. In this application the AFM is related to a much less sensitive device described by Tabor and Winterton (ref. 5) and developed by Israelachvili and coworkers (ref. 6). In contrast to STM, which essentially can only be used with electrical conductors, AFM can also be used with insulators.

In the following sections we discuss optical methods, mainly interferometry, of sufficient resolution to measure AFM deflections. They have an advantage over STM as a metrological device, in that they provide absolute distance, i.e., they do not require calibration by a different procedure.

REVIEW OF OPTICAL METROLOGY WITH ULTRAHIGH SENSITIVITY

Optical Resonators

In the early 1970's much effort was expended to detect the existence of gravity waves. For this purpose various methods capable of measuring very small displacements were explored. One of the best was that of Boersch and coworkers (ref. 7), which claims ability to measure length shifts down to 10^{-5} Å. Figure 1 shows the experimental setup schematically. Its basic element is a three-mode He/Ne laser for which the frequency of one axial mode can be tuned independently from the other modes by a third, partially reflecting and partially transmitting mirror. The coupled resonator thus produced causes a frequency shift which is proportional to the change of the optical path length between the third mirror and the neighboring laser mirror. By observing the beat frequency of the tube, optical path variations of 10^{-5} Å are measurable.

For this system to be used with an AFM the "third" mirror must be coupled to the cantilever. This should not be difficult. A piezoceramic translator moves this mirror. Frequency modulation techniques reduced the noise. The laser must be well stabilized by a servo loop.

Laser Interferometer of Moss, Miller, and Forward

This instrument was also designed for gravity wave detection (ref. 8). The principal difference between it (fig. 2) and the classical two-beam Michelson interferometer is the piezoelectric driver on one of the interferometer mirrors. This driver generates subangstrom (3×10^{-14} m) vibrations of known amplitude. The slightly asymmetric configuration prevents reflected energy from reentering the laser. It also provides two outputs so that two photodetectors can be used in a balanced bridge to reduce signals due to laser amplitude noise. The output of the matched detector bridge is amplified with a tuned amplifier and then mixed with the driving signal in a multiplier to transform the signal frequency down to dc. The signal is then passed through a 2.5 Hz bandwidth low pass filter that determines the system bandwidth. This signal is then squared to give an output proportional to power and recorded. A displacement sensitivity of 3.5×10^{-16} m was attained.

For use with the AFM the Michelson Mirror on top of figure 2 would be coupled to the cantilever. The distance of mirror travel corresponding to two successive maxima of output signal would be equal to half a laser wavelength and smaller distances would be linearly proportional to that distance. The entire system, interferometer and AFM, would be mounted on an optical, vibration isolation table.

Two Frequency Laser Interferometer

The single frequency laser interferometer has two major drawbacks which subsequent design could correct: (1) very critical mirror alignment and (2) dependence of the triggering level of the photon detector on the intensity of the light beam and therefore on intensity variations of the laser source. The first difficulty was corrected by the substitution of cube corners (sometimes called corner cubes) for the mirrors, and the second by the use of a two frequency laser (ref. 9). The cube corners have the characteristic of reflecting beams parallel to their angle of incidence regardless of how accurately they are aligned to the beam. An excellent example of the two frequency laser interferometer is the optical heterodyne profilometer of Sommargren (ref. 10). As the name indicates this instrument was designed to measure surface profiles. It does this with a height sensitivity of 1 Å, but should be readily adaptable to displacement measurements, for example by making use of a reference surface vibrating axially on a piezoelectric mount. Sommargren created modulation by rotating his samples on a table and used the position on the surface traversed by the axis as his reference. Two orthogonally polarized beams of slightly different frequency were focused on the surface to be measured. A magnetic field on the laser source produced these beams by the Zeeman effect. A slight lateral displacement was effected by a Wollaston prism. The phase of the beat frequency of the interfering return beams is directly proportional to the axial displacement between the sample and reference locations.

Multiple-Beam Interferometry (MBI)

Suppose parallel monochromatic light impinges on and traverses two parallel reflecting surfaces at a slight angle. Because of interference between the straight-through and the twice reflected beams, which is delayed in phase, a circular fringe pattern can be observed in the focal plane of a lens on a screen parallel to the surface. The optical arrangement (fig. 3) can be considered a modification of the classical Newton's rings experiment. Fabry and Perot used such an instrument to measure the wavelengths of arc radiations with extreme accuracy. For that purpose the reflectivity (giving rise to multiple beams) of the surfaces had to be made very high, e.g. by silvering, for then the sharpness (finesse) of the fringes increases dramatically. Tolansky (ref. 11) applied this principle in the converse mode. He adapted it to find steps on surfaces by examining the change in intensity near the peak of an interference maximum. In the neighborhood of half the peak intensity a step height (fig. 4) of 3 Å is easily observable, indeed visually observable, with radiation of the green (λ = 5460 Å) mercury line. To quote Tolansky (ref. 12): "..a...noteworthy way of indicating the extreme sensitivity of this optical system is the fact that a single lattice spacing in mica (20 Å) makes no less than a 50 percent alteration in transmitted intensity!"

Since MBI is already so sensitive by ordinary observation, phase-lock detection with electronic detectors and minute displacements by piezocrystals should make it at least an order of magnitude more sensitive.

Evanescent Wave Detection

The use of evanescent wave detection (EWD) for the measurement of very small displacements has not been mentioned in the literature to our knowledge, although work described for other objectives would seem to make EWD a logical candidate. It is particularly appropriate here, for it can be considered optically equivalent to STM.

Electromagnetic theory (ref. 13) states that a plane wave propagating through an optically denser medium (suffix 1) and impinging on the interface separating an optically rare medium (suffix 2) with an incidence angle θ_i (taken from the normal) will be totally reflected if θ_i is larger than the critical angle $\theta_c = \sin^{-1}(n_2/n_1)$. Part of the incident energy will nevertheless penetrate the rarer medium in the form of an evanescent disturbance (sometimes called an "evanescent wave"). The energy distribution along the z-axis, normal to the interface, decays exponentially according to

$$I(z) = I_0(\lambda)e^{-(z/\Lambda)} \tag{1}$$

The characteristic penetration depth, Λ, varies with the incidence angle, θ_i, as

$$\Lambda = \frac{\lambda_0}{4\pi}\left(n_1^2\sin^2\theta_i - n_2^2\right)^{-1/2} \tag{2}$$

where λ_0 is the wavelength of light in vacuum. Λ is independent of the polarization of the electromagnetic wave.

The "evanescent" disturbance that travels into the rarer medium can be found in different ways, one of them by putting a second parallel surface within the decay distance of the first boundary. The evanescent wave then gives rise to a real wave in the second medium, which can be observed (fig. 5). This is the classical method of "frustrating" the reflection (ref. 14). The electromagnetic wave is said to "tunnel" across the gap, analogous to electron tunneling in the STM. Another analogy with STM is the exponential dependence of intensity on gap width given by equation (1). The determination of the evanescent energy is therefore an optical analog to STM as a distance-measuring method or, more to the point perhaps for measuring the lever deflection in the AFM, a method of determining angular displacement in accordance with equation (2). It should be noted that both of these equations are approximate, but sufficient for our purposes. A summary of recent work on these equations is given by Harrick (ref. 15).

A most sensitive technique of coupling to the evanescent wave is by fluorescence (ref. 16). If medium 2 consists of an aqueous solution of fluorescein in low enough concentration to avoid quenching by intermolecular interaction, the fluorescence picked up by a photomultiplier is a very sensitive function of angle (eq. (2)). Harrick (ref. 17) presents tables to show this dependence for attainable indices of refraction. Figure 6 contains a schematic diagram of an angular displacement detector based on the fluorescence excited by the evanescent wave.

EXPERIMENTAL

Atomic Force Microscope Using Optical Interference to Detect Lever Deflection

Figure 7 contains a schematic diagram of the atomic force microscope, according to McClelland and coworkers (ref. 4), to which reference was made in an earlier section of this paper. To sense forces of the order of 10^{-8} N it is necessary to achieve a resolution of ~0.1 nm (1 Å) perpendicular to the surface for levers of reasonable spring constant. Binnig and coworkers (ref. 2) achieved this and they also achieved a resolution of ~10 nm parallel to the surface.

Our experimental effort was limited to obtaining the vertical resolution. The deflection of the lever was to be measured while moving the sample along x and y to scan its surface at a distance close to the tip. For this purpose piezoelectric translators are necessary.

A recent article by Atherton (ref. 19) reviews the state of the art of the piezoelectric translators. Most of today's models operate at low voltage (<1 kV). They are very stiff and can move fast and with great force and dissipate very little heat. Positioning to better than 1 nm is now possible with a linearity of 0.1 percent and a response time of less than 0.5 msec with no detectable hysteresis for small displacements.

Laser Interferometer for AFM

Figure 8 shows schematically our laser interferometer for the AFM. Radiation from the He/Ne laser is split by the beamsplitter cube into a part that falls on a screen for visual alignment or on a photocell for possible laser stabilization and into another part that traverses the optical reference flat and is then focused by the lens onto the reflector (cantilever beam or other mechanical element whose small deflection is to be determined). In the drawing the reflector is attached to a piezoelectric tube whose characteristics are to be determined. After reflection the beam is returned to the beamsplitter and split again into (1) a portion that barely bypasses the laser and falls on a screen where the interference fringes generated by interference with the portion of the beam originally reflected by the reference flat can be observed and (2) a portion that passes the aperture and is ultimately detected by the photodiode. As pointed out in the discussion of the Moss et al. (ref. 8) laser interferometer, the laser radiation should not be reflected back into the laser cavity.

The optical system shown is a combination of the laser interferometer of Moss et al. (ref. 8) and the multiple-beam interferometer of Tolansky (refs. 11 and 12) and others. All the optics are of 1 cm or somewhat larger diameter. The lens is a 10 cm focal length (10X) biconvex lens and all the optical surfaces are flat to 0.1 wavelength or better. The optical flat is antireflection-coated on one side. All the components are rigidly mounted on a steel plate, which sits on an optical table.

The electronic system (fig. 9) was assembled entirely from commercial parts. Differential amplifiers isolate the relatively high voltage necessary

to drive the piezoelectric translators from the dc power supply and the lock-in amplifier. A transformer can be substituted for the differential amplifier DA1, i.e., the one that transfers the oscillating signal, to enhance the signal-to-noise ratio. The lock-in's reference oscillator also supplied the modulation frequency of 5000 Hz to the piezoelectric translators. Either the reflector or the optical reference flat can be vibrated; however, it turned out to be preferable to vibrate the reference flat. The need for the vibrating of a component arises from ac detection with the lock-in amplifier, which is the standard method of detection for small signal-to-noise ratios.

The 5000 Hz oscillation frequency was arbitrarily chosen because of its audibility. However, it turned out to be rather high for the time constant of the crystal. A 500 Hz frequency gave better signal-to-noise ratios.

The laser interferometer (fig. 8) measures the changes of distance between the optical reference flat and the reflector. Because of the interposed lens the interference pattern intercepted on the screen next to the laser or in the plane of the photodiode consists of (Newton's) rings. Distance changes (displacements) can be determined from changes in the ring radii or, more accurately, from changes of ring intensity at say half height (Fig. 4) recorded photoelectrically.

RESULTS

Figures 10 and 11 show photographs of the ring patterns for two slightly different positions of the reflector. Figure 12 shows a recorder trace for a time constant of four seconds. It was found that the innermost fringe of figure 10 moved by half a fringe when a dc potential of 300 V was put on the piezoelectric translator (a tube). The noise level on the chart was 0.5 units, corresponding to 0.7 V applied to the piezoelectric translator. Since half a fringe corresponds to approximately 1500 Å of displacement, a displacement sensitivity of

$$\psi = 1500\left(\frac{0.7}{300}\right) = 3.5 \text{ Å (for time constant 4 sec)}$$

$$= 2.0 \text{ Å (for time constant 12.5 sec)}$$

was calculated. When the time constant was set at 12.5 sec on the lock-in amplifier, the corresponding ψ was about 2 Å. This value is about twice that of Binnig (ref. 3), but should still be adequate for the purpose. It would very likely have been reduced by half if the modulation frequency had been 500 instead of 5000 Hz. However, lack of time prevented further testing at that stage of the program.

DISCUSSION AND CONCLUSION

It seems clear that the main reason for attaining an accuracy and precision of measurement on the level of angstroms was the piezoelectric positioners, for the laser was not stabilized and the optical table was of inadequate quality. Tolansky had paid considerably more attention to detail, but reached only a sensitivity of about 3.5 Å. The optical table could be improved by a

heavier table top. However, laser stabilization would probably be more important. The best way of doing this would be the use of two laser frequencies from the same laser source in a way similar to Sommargren's method referred to earlier. Intensity variations are thus immediately compensated. Time did not permit construction of a three-mode laser system similar to that of Boersch (ref. 7), which was discussed. It is questionable, however, whether an AFM even in a vacuum could be made stable enough to benefit from it. On the other hand, the evanescent wave/fluorescence system could be made much simpler than our laser interferometer and would be potentially more precise. It is currently under study.

It is worth repeating that optical metrology is capable of angstrom or subangstrom accuracy and precision only in the axial (z-) direction. To get high lateral accuracy and precision the very fine stylus, perhaps of atomic dimensions, used in the STM and AFM is needed as a probe.

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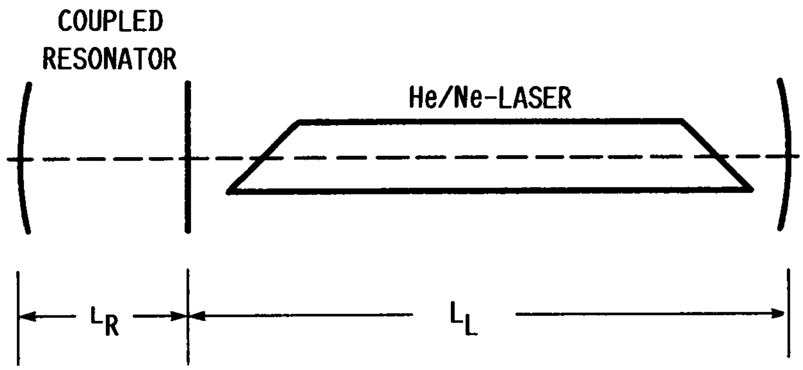

FIGURE 1. - LASER WITH COUPLED RESONATOR.

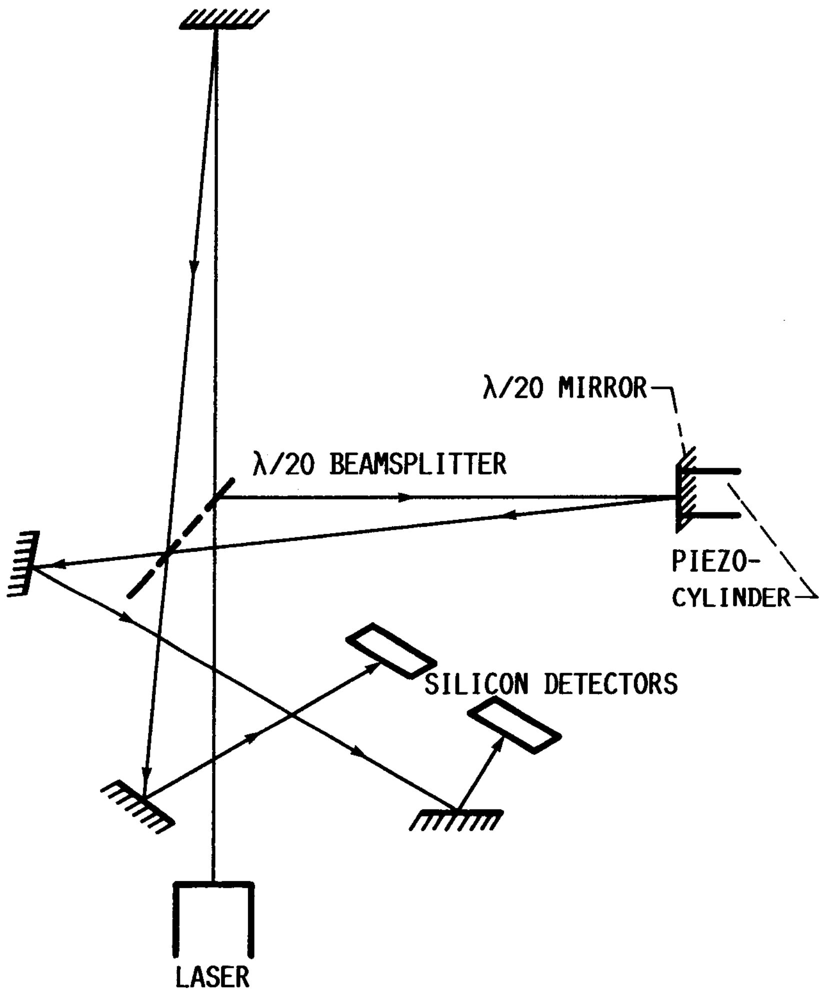

FIGURE 2. - MICHELSON INTERFEROMETER OF MOSS ET AL. [8].

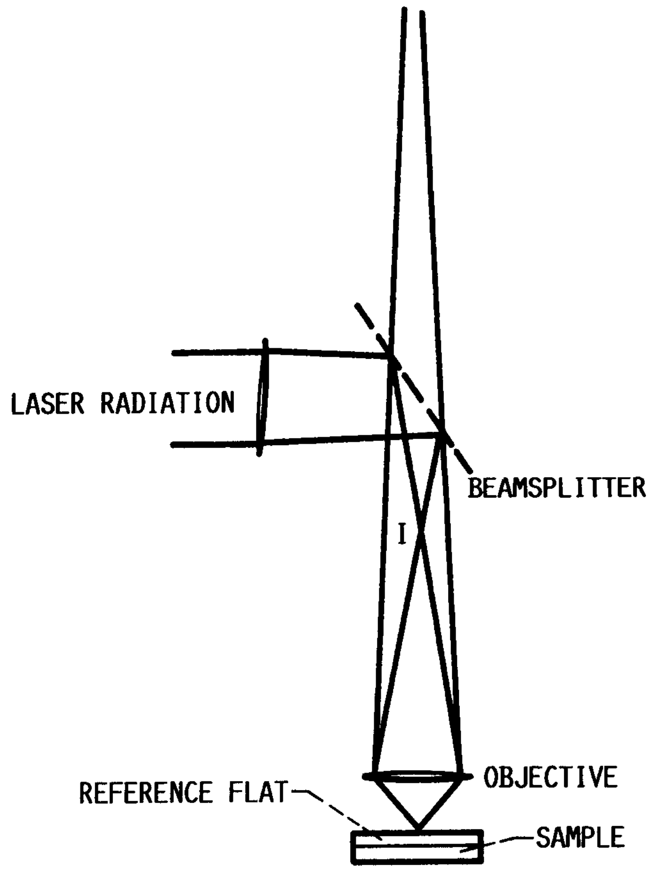

FIGURE 3. - TOLANSKY'S SETUP FOR MULTIPLE-BEAM INTERFEROMETRY.

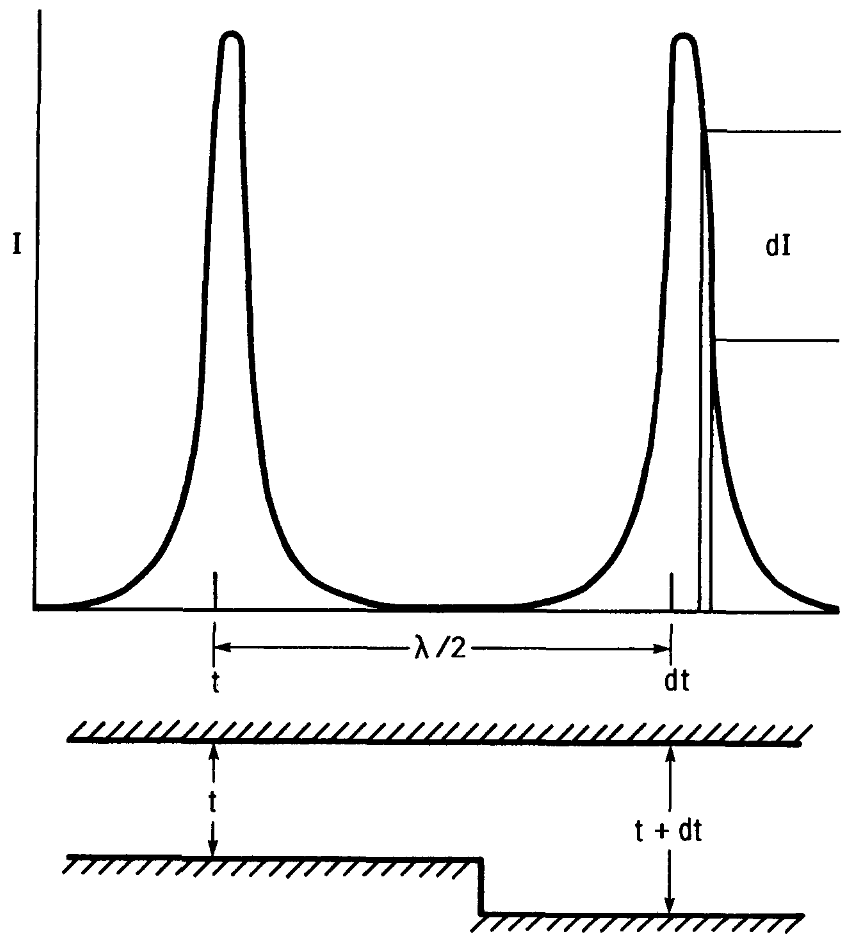

FIGURE 4. - VIEWING WITHIN A SHARP FRINGE GIVES A LARGE INTENSITY CHANGE dI FOR A SMALL STEP dt BETWEEN REFLECTORS.

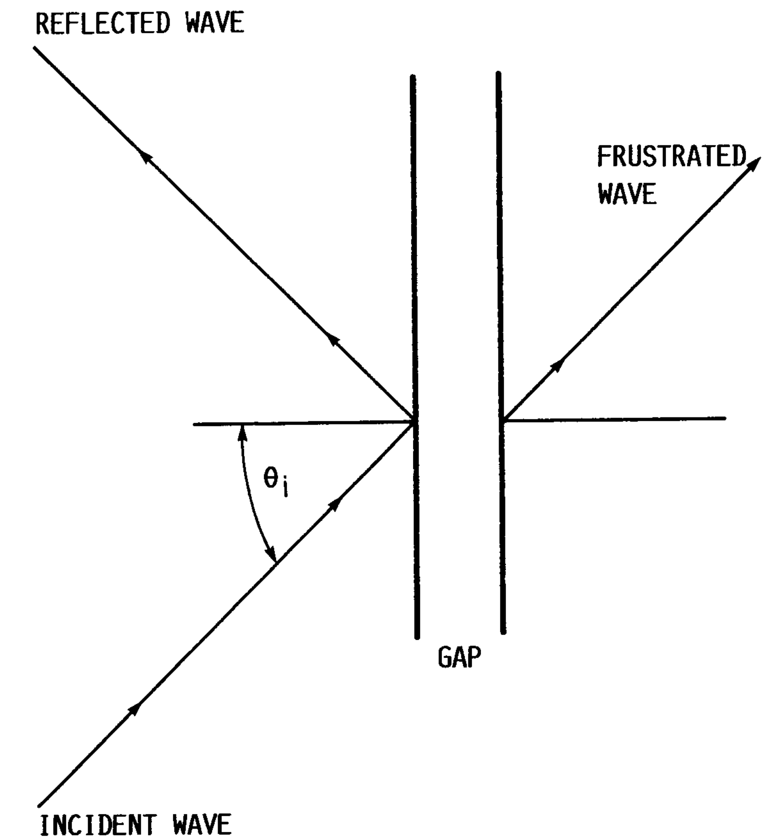

FIGURE 5. - FRUSTRATION OF THE EVANESCENT WAVE ($\theta_i > \theta_c$).

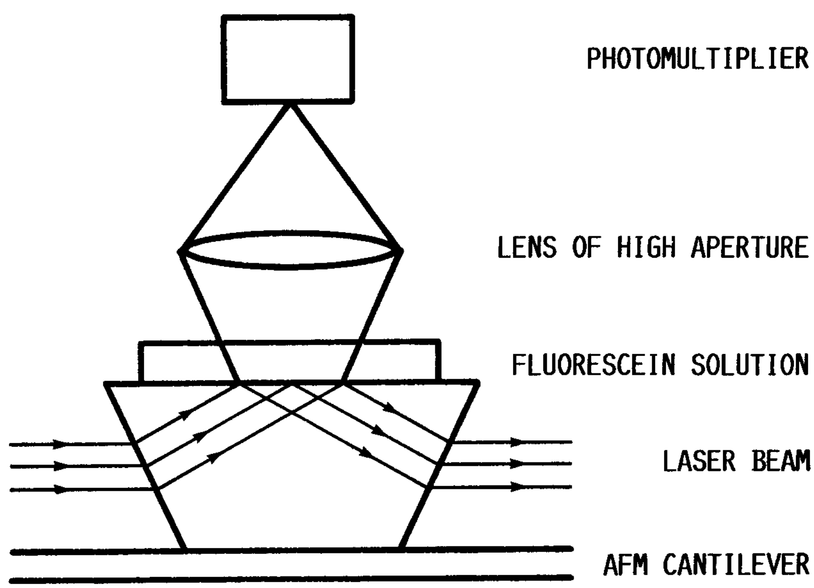

FIGURE 6. - EVANESCENT WAVE-EXCITED FLUORESCENCE AS AN ANGULAR DISPLACEMENT DETECTOR.

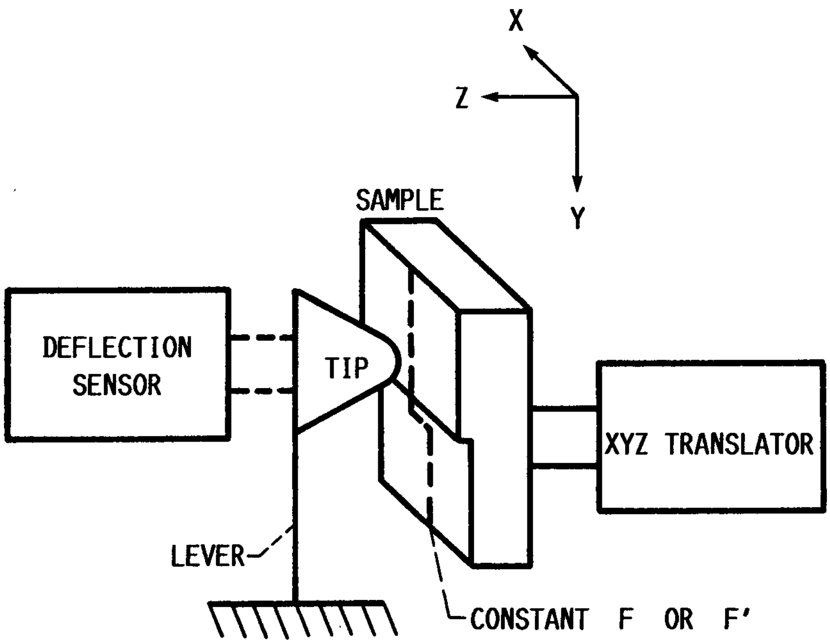

FIGURE 7. - ATOMIC FORCE MICROSCOPE (SCHEMATIC).

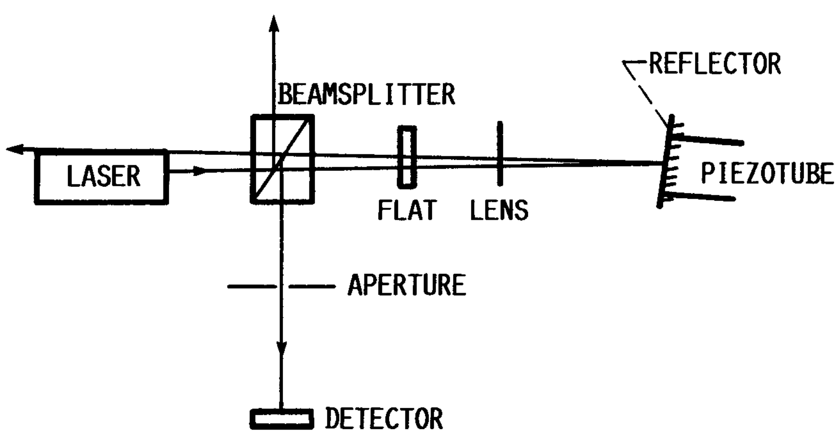

FIGURE 8. - LASER INTERFEROMETER USED IN THIS STUDY: OPTICS.

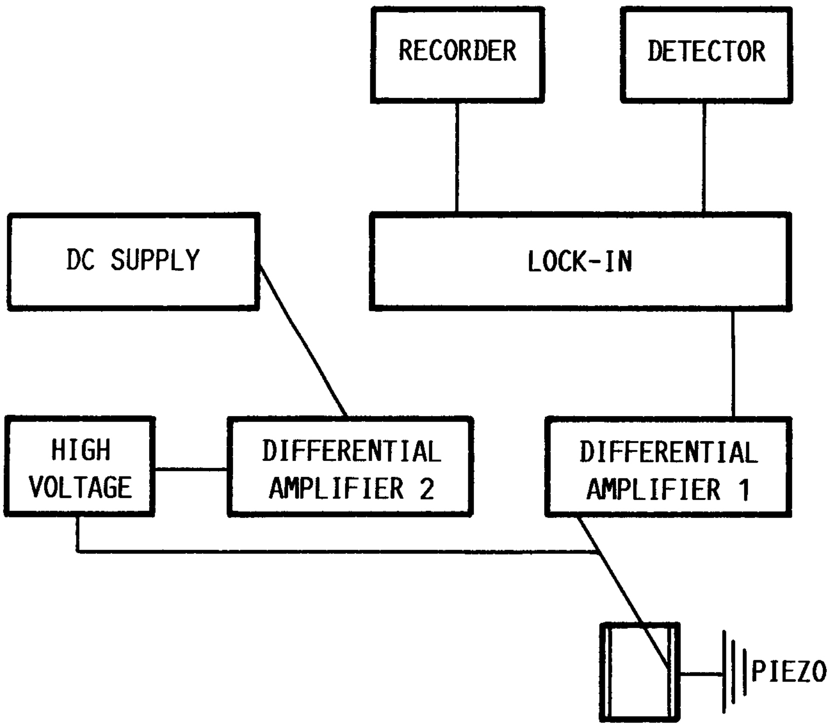

FIGURE 9. - LASER INTERFEROMETER USED IN THIS STUDY: ELECTRONICS.

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OF POOR QUALITY

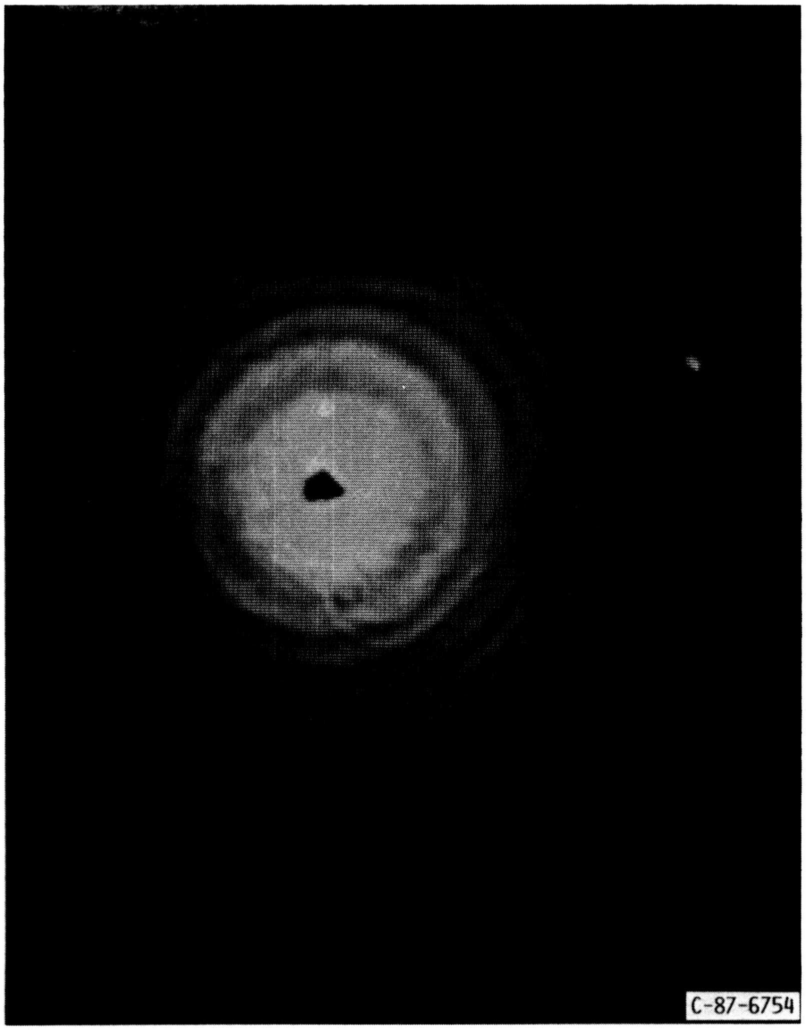

FIGURE 10. - INTERFERENCE RINGS OF THE LASER INTERFEROMETER (ORIGINAL POSITION OF REFLECTOR).

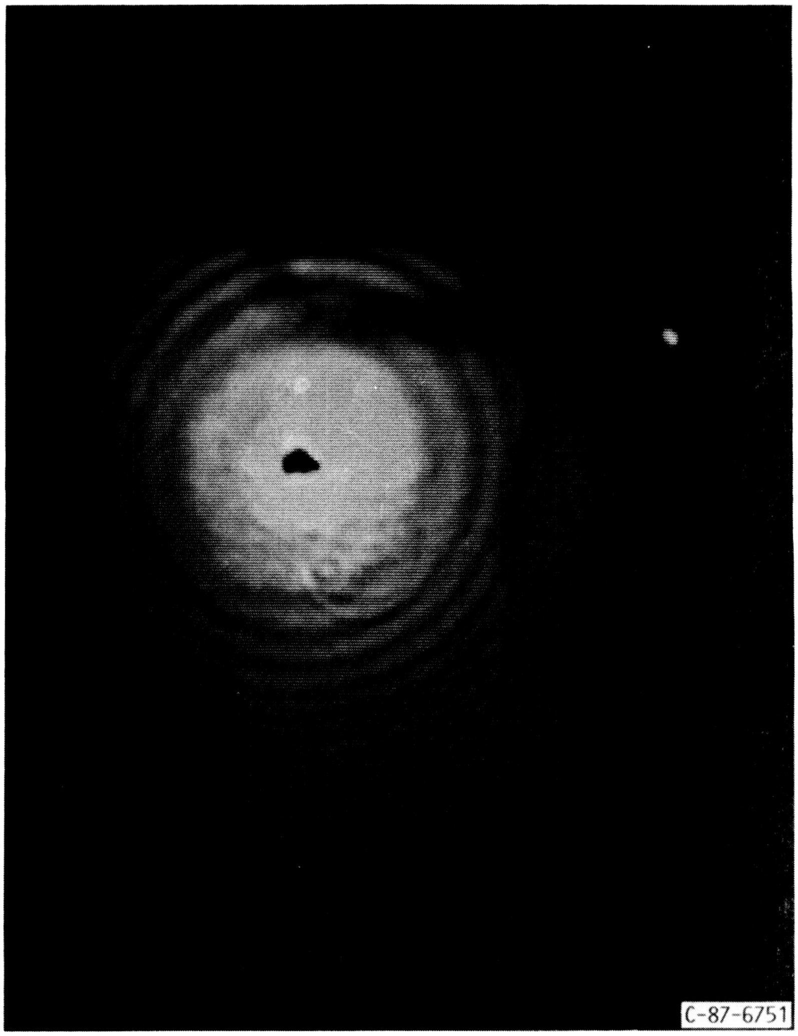

FIGURE 11. - INTERFERENCE RINGS OF THE LASER INTERFEROMETER (SLIGHT DISPLACEMENT OF REFLECTOR). THE CHANGE IN INTENSITY OF THE SMALLEST RING AND THE CHANGE OF RADIUS OF THE NEXT ONE ARE CLEARLY OBSERVABLE.

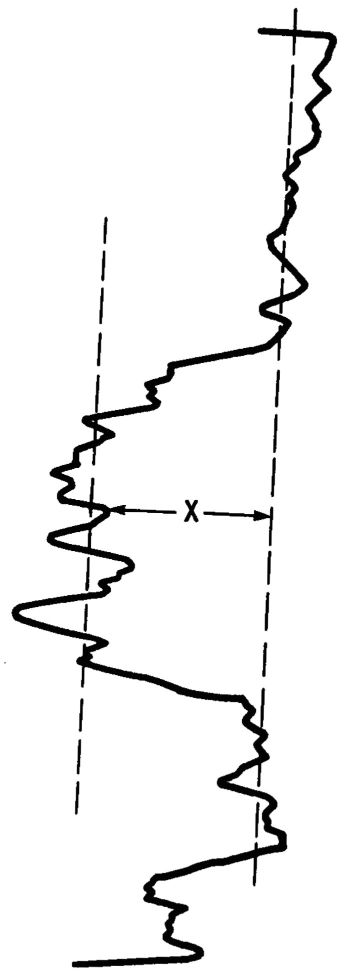

FIGURE 12. - RECORDER TRACE FOR A 7-VOLT CHANGE (X = 7 V) OF THE POTENTIAL ON THE PIEZO TRANSLATOR. NOTE THAT THE NOISE LEVEL IS ABOUT ONE TENTH OF THAT OR 0.7 VOLT. THE TIME CONSTANT WAS 4 SECONDS. THERE WAS A SLIGHT THERMAL DRIFT.

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16. Abstract

The recent development of the scanning electron tunneling microscope and the atomic force microscope requires absolute standards for measurements in the angstrom and subangstrom range. Optical interferometry with lasers and multiple mode laser resonances can provide absolute measurements as the laser wavelengths are very accurately known. A key feature of such measurements is the use of piezoelectric crystals as translators of the highest accuracy for very small distances. However, the dimensional changes of these crystals resulting from electrical potential changes depend on many variables, among them the method of mounting, so that accurate calibrations are necessary. Starting from advances in optical metrology made by physicists trying to find gravity waves, advances which led to measurements down to 10^{-5} Å, the author designed and built a much simpler system for the angstrom range. The major limiting factors were mechanical vibrations, air currents, thermal changes and laser instabilities.

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